

We claim

1. A process for the preparation of (+)2-(4-chlorophenyl)-3-methyl butanoic acid which comprises reacting (\pm)2-(4-chlorophenyl)-3-methyl butanoic acid (CPA) with a resolving agent comprising an amine in a hydrophobic/hydrophilic organic solvent in the presence
5 of water, separating the desired amine salt and refining the salt with the same solvent system used for resolution and recovering the desired (+)CPA and undesired (-)CPA and amine resolving agent.
2. A process as claimed in claim 1 wherein the resolution is conducted by treating racemic CPA with an amine to precipitate a salt of one enantiomer of CPA.
- 10 3. A process as claimed in claim 1 wherein the solvent is selected from the group consisting of an aliphatic, cycloaliphatic, aromatic hydrocarbon, hydroxylic solvent and any mixture thereof.
4. A process as claimed in claim 3 wherein the solvent is selected from the group consisting of methanol, ethanol, propanol, isopropanol, butanol, iso-butanol, tert-butanol, toluene
15 and any mixture thereof.
5. A process as claimed in claim 4 wherein the solvent is selected from the group consisting of butanol, propanol, water and any mixture thereof.
6. A process as claimed in claim 1 wherein the amine resolving agent is an optically active amine.
- 20 7. A process as claimed in claim 6 wherein the optically active amine is an arylamine containing 8 to 20 carbon atoms.
8. A process as claimed in claim 7 wherein the arylamine is selected from the group consisting of α -phenyl- β -(p-tolyl)ethylamine, α -phenyl- β -phenylethylamine, α -phenylethylamine and N,N-dialkyl α -phenylethylamine.
- 25 9. A process as claimed in claim 7 wherein the arylamine is selected from the group consisting of N,N dimethyl, N, N diethyl, N,N dipropyl, N,N diisopropyl, N-methyl, N-ethyl and higher alkyl amines.
10. A process as claimed in claim 7 wherein the aryl amine is (S)(-) α -phenylethylamine.
11. A process as claimed in claim 1 wherein the amine resolving agent is used in amount of
30 0.4 to 0.65 mole per mole of (\pm)CPA.
12. A process as claimed in claim 1 wherein the amine is added in neat form or in the form of solution.
13. A process as claimed in claim 1 wherein the amine is added in one lot or over a period of time ranging from 10-60 minutes.

14. A process as claimed in claim 1 wherein the amine is added at a temperature in the range of 30 to 100°C.
15. A process as claimed in claim 1 wherein the amine is added to the racemic CPA solution.
16. A process as claimed in claim 1 wherein the racemic CPA solution is added to the amine.
- 5 17. A process as claimed in claim 1 wherein the solvent used is in the range of 20-40% as aqueous solution and two to three times by weight based upon the amount of CPA used.
18. A process as claimed in claim 1 wherein the resolution reaction is carried out over a period of 2 to 6 hours.
19. A process as claimed in claim 1 wherein the amine salt formed is substantially in the form
10 of a precipitate.
20. A process as claimed in claim 1 wherein the temperature range during separation of optically active salt is in the range of ambient temperature to 80°C.
21. A process as claimed in claim 1 wherein the crystallized salt is separated by filtration or centrifugation.
- 15 22. A process as claimed in claim 1 wherein the optically active amine salt obtained is refined in a hydrophilic solvent selected from the group consisting of methanol ethanol, propanol, isopropanol, butanol, 2-butanol, tert butanol and an aqueous mixture thereof.
23. A process as claimed in claim 22 wherein the hydrophilic solvent is selected from the group consisting of butanol, propanol and an aqueous mixture thereof.
- 20 24. A process as claimed in claim 1 wherein the optically active salt is refined at a temperature ranging from 40 to 120°C.
25. A process as claimed in claim 24 wherein the solvent used for refinement is in the range of 20-40% as aqueous solution and one to four times by weight based on the amount of optically active salt used.
- 25 26. A process as claimed in claim 24 wherein the duration of refinement is in the range of 3-5 hrs.
27. A process as claimed in claim 24 wherein the optically active salt is separated after refinement at a temperature in the range of 40 to 70°C.
28. A process as claimed in claim 24 wherein the optically active salt after refinement is
30 separated by filtration or centrifugation.
29. A process as claimed in claim 1 wherein the optically active salt of (+) CPA after refinement is liberated using a mineral or an organic acid.
30. A process as claimed in claim 29 wherein the mineral acid used for liberation of optically active acid is selected from hydrochloric acid and sulphuric acid.

31. A process as claimed in claim 30 wherein the mineral acid is aqueous sulphuric acid.
32. A process as claimed in claim 29 wherein the aqueous mineral acid layer containing amine salt is combined with aqueous mineral acid layer obtained from recovery of the undesired (-) CPA.
- 5 33. A process as claimed in claim 1 wherein the mother liquor enriched with undesired (-) CPA salt obtained after precipitating the desired (+) CPA salt is concentrated at reduced pressure for recovery of (-) CPA.
34. A process as claimed in claim 1 wherein the undesired (-) CPA salt after concentration is treated with aqueous mineral/organic acids and extracted with hydrophilic/hydrophobic organic solvents and concentrated under reduced pressure for obtaining (-) CPA.
- 10 35. A process as claimed in claim 34 wherein the mineral acid used for liberation of (-) CPA from its amine salt is selected from hydrochloric acid and sulfuric acid.
36. A process as claimed in claim 35 wherein the mineral acid is aqueous sulfuric acid.
37. A process as claimed in claim 34 wherein the liberated acid is treated with an organic solvent selected from dichloromethane, dichloroethane, chloroform, toluene and hexane.
- 15 38. A process as claimed in claim 34 wherein the liberated acid is treated with an organic solvent comprising toluene.
39. A process as claimed in claim 34 wherein the aqueous mineral acid layer containing amine salt is combined with the corresponding aqueous mineral acid layer obtained from the liberation of desired (+) CPA to effect the recovery of optically active resolving agent.
- 20 40. A process as claimed in claim 39 aqueous mineral acid layers obtained from liberation of (+) CPA and (-) CPA are mixed, cooled preferably to 10 to 5°C and extracted with aqueous caustic lye solution of concentration ranging from 20-80% to recover the resolving amine employed in resolution of (\pm) CPA.
- 25 41. A process as claimed in claim 40 wherein the concentration of the aqueous lye solution is in the range of 30-60%.
42. A process as claimed in claim 1 wherein the crude amine obtained is used in subsequent batches of (\pm) CPA and the alkaline layer is extracted with an hydrophilic/hydrophobic organic solvent selected from the group consisting of benzene, toluene, hexane, dichloromethane, dichloroethane and chloroform.
- 30 43. A process as claimed in claim 42 wherein the solvent is selected from benzene, toluene and hexane.
44. A process as claimed in claim 42 wherein the solvent is toluene.